

DOCKET NO: 288922US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
ROBERT OWEN, ET AL. : EXAMINER: WAYNE LANGEL  
SERIAL NO: 10/574,289 :  
FILED: JANUARY 16, 2007 : GROUP ART UNIT: 1793  
FOR: PROCESS FOR THE :  
PURIFICATION OF AQUEOUS  
PEROXYGEN SOLUTIONS, SOLUTIONS  
OBTAINABLE THEREBY AND THEIR  
USE

DECLARATION

Now comes Jurgen Bosse, who deposes and states as follows:

1. That I am an inventor of the above-identified U.S. patent application, and that I understand English.

2. That I understand that pending Claim 1 of the above-identified U.S. patent application reads:

1. A process for the purification of an aqueous hydrogen peroxide solution comprising the treatment of an aqueous hydrogen peroxide solution first with a reverse osmosis membrane purification, and afterwards with at least one other purification selected from the group consisting of exposure to ultraviolet light, exposure to ozone, contact with at least one adsorption resin, and any combination thereof

3. That I understand that the U.S. Patent Office has taken the position that pending Claim 1 is obvious when exposure to ozone is not required because the Office believes that there is no evidence on record of unexpected results which emanate from a combination of purification steps in the order as recited in pending Claim 1 as opposed to the reverse order.

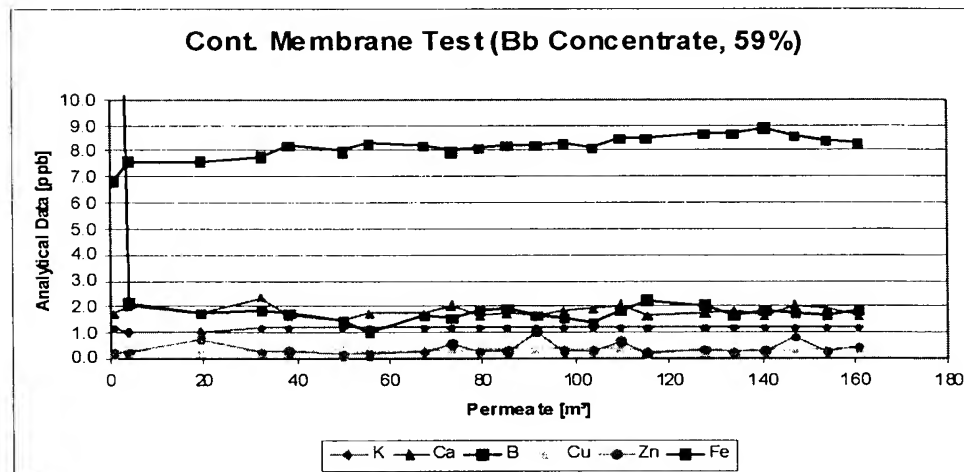
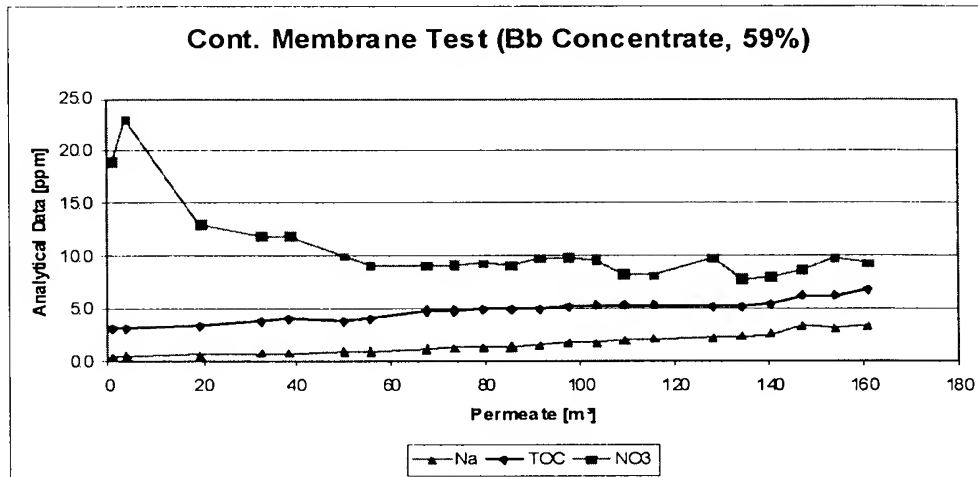
4. That unexpected results do in fact emanate from a combination of purification steps in the order as recited in pending Claim 1 as opposed to the reverse order.

5. That in studying the purification of aqueous hydrogen peroxide solutions **my co-inventors and I** discovered that, surprisingly, reverse osmosis (RO) membranes in fact contribute certain impurities, such as  $\text{NO}_3$ , to the aqueous hydrogen peroxide solution being purified. To my knowledge this was totally unexpected.

6. Aqueous hydrogen peroxide is an important raw material for the semiconductor and microelectronics industries, in which it is used in many of the processing steps that result in the finished semiconductor or component. The industry demands a high level of purity from the hydrogen peroxide solutions. Cationic and anionic contamination of the semiconductor process results in lowered production yields and therefore higher costs. Thus, purification beyond distillation is required to produce a semiconductor grade material.

7. Given this demand within the electronics industry and our unexpected finding that reverse osmosis (RO) membranes in fact contribute certain impurities, such as  $\text{NO}_3$ , to the aqueous hydrogen peroxide solution being purified, the order of steps as claimed herein - RO membrane treatment first followed by at least one of exposure to ultraviolet light, exposure to ozone, and contact with an adsorption resin - is critical to providing a high purity aqueous hydrogen peroxide solution product that is suitable for use in, e.g., semiconductor processing. Providing the purification steps in reverse order to that presently claimed would leave the impurities contributed by the RO membrane in the solution. Thus, the order of steps in pending Claim 1 provides a quite different, and unexpected, result as compared to the same steps in reverse order.

8. That **my co-inventors and I** conducted tests on an RO membrane to determine the type and amount of impurities contributed by the RO membrane. The results of these tests are provided below:



9. These tests were conducted as follows:

A new reverse osmosis membrane (8" membrane module SWC line of Hydranautics ) was flushed with de-mineralized water, and then the reversed osmosis system was switched to 59% hydrogen peroxide feed. The hydrogen peroxide permeate flow was sampled and analyzed for

cationic and anionic impurities as well as for organic carbon (TOC). All analyzed parameters were found to reach relatively stable concentration levels after a few cubic meters of permeate had been produced, but we found different results for nitrate (NO<sub>3</sub>). This impurity was released from the reversed osmosis membrane for a longer period of time and when it finally stabilized it did so at a higher level compared to other impurities (see graphs above). From these results it became obvious to us that the reverse osmosis membrane itself contributes to remaining impurity level of the permeate, and at least one secondary purification step has to be added AFTER the reversed osmosis membrane treatment.

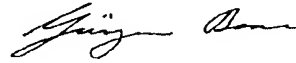
These tests and the results obtained show, e.g., NO<sub>3</sub>, being added to the aqueous hydrogen peroxide solution being purified by the RO membrane, which was totally unexpected.

10. That from these tests it is clear that unexpected results do in fact emanate from a combination of purification steps in the order as recited in pending Claim 1 as opposed to the reverse order, because we have surprisingly found that reverse osmosis (RO) membranes in fact contribute certain impurities, such as NO<sub>3</sub>, to the aqueous hydrogen peroxide solution being purified. To my knowledge this was totally unexpected, and thus the order of steps as claimed herein provides a quite different, and unexpected, result as compared to the same steps in reverse order.

11. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section

1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

12. Further deponent saith not.



27<sup>th</sup> May 2008

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Jurgen Bosse

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DATE